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Supercritical Fluid Extraction of Organochlorine Pesticides Using Trifluoromethane

Scott L. Taylor and Jerry W. King

Food Quality and Safety Research
National Center for Agricultural Utilization Research
USDA/ARS, Peoria, IL 61604

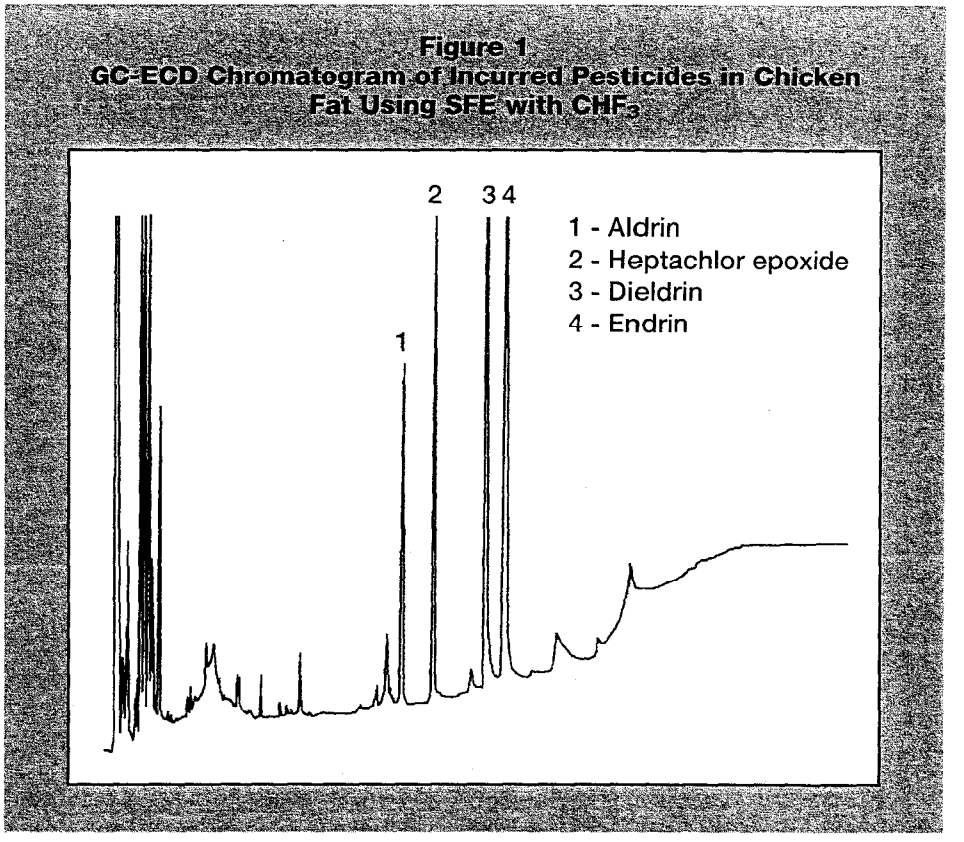
Objective:

Analysis of trace levels of pesticides in food and agricultural products is often complicated by the presence of coextracted fat or lipid material. These interfering coextractives often result in the need for extensive sample cleanup prior to chromatographic analysis. Supercritical fluid extraction (SFE) with trifluoromethane (halocarbon-23, CHF_3) provides an excellent way of recovering the organochlorine pesticides without the attendant problem of coextracted fat. In this study, the extraction of three organochlorine pesticides from poultry adipose tissue is described.

Experimental:

The incurred analytes were extracted with CHF_3 (Air Products and Chemicals, Inc., Allentown, Pa.) from poultry adipose tissue utilizing an Isco (Lincoln, Nebr.) SFX 2-10 supercritical fluid extractor. The rendered poultry tissue (0.20–0.22 g) was pipetted onto a glass bead support (3.1 g, 40–50 mesh) contained in a 2.5 mL extraction cell. Extractions were performed at 250 atm and 50°C using 50 mL CHF_3. Analyte collection was accomplished in a test tube containing 10 mL of hexane. One set of extracts was directly analyzed by GC-ECD after concentration to 0.5 mL under a N_2 stream and addition of an internal standard (0.5 mL). A second set of extracts was gravimetrically analyzed for the amount of extracted fat. A typical ECD chromatogram of the direct extract is shown in *Figure 1*. Aldrin was used as an internal standard in the assay.

Figure 1
GC-ECD Chromatogram of Incurred Pesticides in Chicken Fat Using SFE with CHF_3



Results and Discussion:

The peaks numbered 1, 2, 3, and 4 in *Figure 1* are aldrin, heptachlor epoxide, dieldrin, and endrin, respectively, at a 1–3 ppm level in the poultry fat. Although there are spurious peaks in the chromatogram (*Figure 1*), the assay easily permits the quantitation of the three organochlorine pesticides without sample cleanup. It should be noted that the chosen extraction conditions were identical to those used for SC-CO_2 of the same sample matrix[1], in order to permit a direct comparison of the discriminating power of CHF_3 for lipid coextractives.

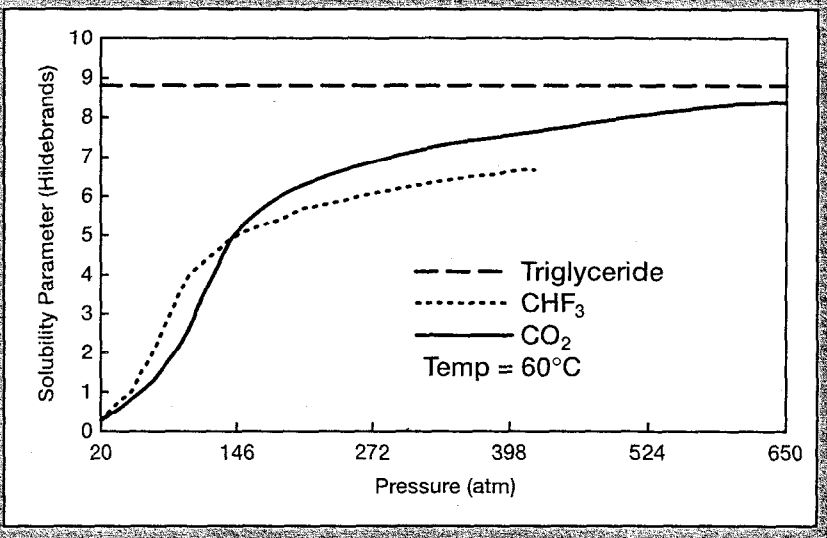
Table 1
SFE of Chicken Fat: CO_2 vs. CHF_3

Matrix support	% Fat Recovery	
	CO_2	CHF_3
Glass beads	54.4	0.45

Table 1 lists the percentage of fat extracted by SC-CO_2 and -CHF_3, respectively, based on the initial charge of adipose tissue in the extraction vessel. There is literally a 100-fold decrease in the amount of fat extracted when using SC-CHF_3 in place of SC-CO_2. This could even be further improved by mixing a selective adsorbent, such as alumina, with the adipose tissue in the extraction cell[1].

A rationale for the above results can be found by invoking the solubility parameter concept to describe the relative solvating power of the two fluids for lipid moieties. As shown in *Figure 2*, the solubility parameter for SC-CHF_3 and -CO_2 are plotted as a function of pressure, relative to the approximate value for the triglyceride molecule, the major constituent of fats. At lower pressures, trifluoromethane is a better solvent for triglycerides than CO_2, by virtue of having a solubility parameter that is closer to that exhibited by the triglyceride moiety. However at 250 atm, the opposite is true, with the SC-CO_2 becoming a better solvent for triglyceride-based fats/oils[2]. This trend has been observed by other investigators[3] and can be used to advantage in natural product extractions.

Figure 2
Solubility Parameter vs. Pressure



References:

1. J. E. France, J. W. King, and J. M. Snyder, Supercritical fluid-based cleanup technique for the separation of organochlorine pesticides from fats. *J. Agric. Food Chem.*, **39:** 1871–1874 **(1991)**.

2. J. W. King, Supercritical fluid extraction of polymers and solvents: Utilization of the solubility parameter concept. *Proceedings of the Division of Polymeric Materials: Science and Engineering – American Chemical Society,* **51:** 707–712 **(1984)**.

3. E. Stahl, K. W. Quirin, and D. Gerard, *Dense Gases for Extraction and Refining,* Springer-Verlag, Heidelberg **(1988)**.

More Information:

If you'd like more information about Air Products' SFC/SFE grade CO_2 or if you'd like copies of other Performance Bulletins, please contact us at the address below.

Air Products and Chemicals, Inc.
Specialty Gas Department
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Phone: 1-800-752-1597